

Tritium In Soil Azeotropic Method

Principle

Tritium activity in the water content of sample is radio- assayed after separation of the water by a cyclohexane distillation. A measured aliquot of the sample is heated in an azeotrope still together with cyclohexane. The separated water is measured and counted for tritium using a liquid scintillation counter.

Sample Preparation

Sample sizes and Quantity of Cyclohexane.

Sample type -----	Sample size -----	Cyclohexane -----
Soil	200 g	1300 ml
Hay	50 g	400 ml
Green chop	30 g	70 ml
Biological tissue	30 g	150 ml
Urine	20 ml	50 ml
Milk	30 ml	70 ml

Equipment

- A. Azeotropic still (figure #1).
- B. Liquid scintillation counter.

Reagents

- A. Cyclohexane, spectrophotometric grade reagent.
- B. Desiccant, calcium chloride type.
- C. Instagel scintillation cocktail.

- D. Low background water.
- E. Tritium standards of approximately 400 dpm, 200 dpm, 100 dpm, and 50 dpm.Procedure
- A. Add measured amounts of the sample and cyclohexane to a distillation flask using the amounts given.
- B. Assemble the distillation apparatus and heat the sample flask until water stops collecting in the receiver.
- C. Stop the distillation and measure the total volume of collected water.
- D. Place a 6 ml aliquot of the collected water in a LS vial (Kimble 25 ml vials) and add 18.5 ml of Instagel.
- E. Prepare a blank by adding low tritium water using the same volume as the sample.
- F. Prepare a standard using the same volume as the sample.
- G. Place the sample vials in the LS counter and allow them to dark adapt for 4-6 hours.
- H. Count the samples for 500 minutes.

Calculations

$$\text{Tritium in pCi/l} = \frac{1000 A}{2.22 E V}$$

A = net cpm of sample
E = efficiency
V = volume of sample

Lower Limit of Detection

$$\text{LLD in pCi/liter} = \frac{(4.66) \sqrt{B(t)}}{\text{-----}} \times 1000$$

$$(2.22)(E)(t)(V)$$

where:

- B = Background cpm
- t = time counted (samples and background should be counted for the same amount of time)
- E = Counting efficiency
- V = Sample volume counted (in ml)

$$2.22 = \text{dpm per pCi}$$

References

Robert Lieberman, EPA Eastern Environmental Radiation Facility Montgomery, Alabama.